

EXHIBIT I

Docket No.: 0425-1218PUS1  
(PATENT)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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In re Patent Application of:  
Tetsuya OKANO et al.

Application No.: 10/551,654

Confirmation No.: 5662

Filed: July 10, 2006

Art Unit: 1616

For: A COMPOSITION FOR PRODUCTION OF A  
STERILIZER AND A PROCESS FOR  
PRODUCING ORGANIC PERACID

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Examiner: A. L. Fisher

**DECLARATION UNDER 37 C.F.R. § 1.132**

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir:

I, Noboru Matsuo, hereby declare as follows:

I am one of the co-inventors of the invention as described and claimed in the above-identified patent application.

I have carried out additional examples myself or under my direct supervision. Test procedures and results are shown below.

**Side-by-Side Comparison between the Present Invention and the Primary Reference**

The Examiner has cited U.S. Patent No. 5,827,447 to Tamura et al. (hereinafter, "Tamura '447") as the primary reference in a rejection under 35 U.S.C. § 103(a). I consider Example 11 of Tamura '447 to be the closest example to the present invention.

Enclosed herewith is Table A, which shows inventive Example 3-3 and Tests 1 and 2 as comparative examples. Test 1 was carried out using the same materials and methods as disclosed for Example 11 of Tamura '447. Test 2 was carried out using the same materials and methods as disclosed for Example 11 of Tamura '447, except triacetine was used in place of NOBS.

The obtained products were evaluated in the same way as Example 3-3 of the present specification. The results of all three examples are shown in Table A.

As shown in Table A, the number of remaining microorganisms with the inventive example is much less than the number with the comparative examples. As such, the present invention provides unexpectedly superior results.

#### **Side-by-Side Comparison between the Present Invention and the Secondary Reference**

The Examiner has cited U.S. Patent No. 5,869,440 to Kobayashi et al. (hereinafter, "Kobayashi '440") as the secondary reference in a rejection under 35 U.S.C. § 103(a). I consider Comparative Example 4 of Kobayashi '440 to be relative to the present invention.

Enclosed herewith is Table B, which shows inventive Example 3-3 and continued Example 3-3 with changed reaction temperatures and reaction times and Test 3 and continued Test 3 with changed storage temperatures and storage terms as comparative examples.

Test 3 was carried out using the same materials and methods as disclosed for Comparative Example 4 of Kobayashi '440, except changed storage temperatures and storage terms.

The obtained products were evaluated in the same way as Example 3-3 of the present specification. The results are shown in Table B.

As shown in Table B, the number of remaining microorganisms with the inventive example is much less than the number with the comparative examples. As such, the present invention provides unexpectedly superior results.

The undersigned declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S. Code 1001 and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

By: Noboru Matsuo Date: August 29, 2010  
Noboru Matsuo

Table A

components	used materials	Example 3-3 of USSN 10/551654	Test 1 Example 11 of Tanura et al	Test 2
Betaain surfactant*			10.0	10.0
(A)	Triacetin	5.0	-	2.0
	NOBS	-	2.0	-
(B)	H2O2	1.5	5.0	5.0
Organic phosphoric acid (Purity)	HEDP**	0.1	-	-
	EDTMP***	-	0.1	0.1
Alkaline pH adjusting agent	NaOH	2.0	-	-
Acidic pH adjusting agent	Phosphoric acid(85%)	5.0	-	-
	Sulfuric acid	-	very small amount	very small amount
Total		110.0	100.0	100.0
(A)/(B) molar ratio		0.52	0.04	0.06
Organic peracid concentration(ppm) after preparation		27000	500	500
pH of aqueous solution for sterilization (25°C)		3.7	2.0	2.0
Number of remaining microorganisms	Bacillus cereus IFO13494	<50	$1.8 \times 10^7$	$1.5 \times 10^7$
	Bacillus subtilis var. niger	<50	$2.6 \times 10^7$	$2.4 \times 10^7$

Note: \* is softazoline LSB. \*\* is Dequest 2010. \*\*\* is Dequest 2046  
 "1.5" as the amount of H2O2 of Example 3-3 is equivalent to "4.3  
 g" of Table 10 of the instant application. "4.3 g" of Table 10 is the  
 amount of the 35 wt.% aqueous solution of H2O2. 4.3 g x 0.35% is  
 equal to 1.5.

Table B

components		USSN 10/551654		Kobayashi et al.	
		Example 3-3		Test 3, Comparative Example 4	
(A)	Triacetin	5.0	the same components as Example 3-3	2.00 **	the same components as Test 3
(B)	H <sub>2</sub> O <sub>2</sub>	1.5		2.75 **	
Organic phosphonic acid	HEDP*	0.1			
Alkali pH adjusting agent	NaOH sodium ortho-silicate	2.0		1.5 **	
Acid pH adjusting agent	85% phosphoric acid	5.0			
Total		110.0		100.00	
(A)/(B) molar ratio		0.52		0.11	
at the first step	Reaction temperature	25°C~33°C	← ← ← ← ←	25°C	← ← ← ← ←
	Reaction time	10 minutes	minutes 1 day 5 days	Just after	120 minutes 1 day 5 days
Storage of each solution	temperature	120			
Condition of Kobayashi	term				
Concentration of peracid after preparation (ppm)		27000	11000 1500 150	13000	190
pH of aqueous solution for sterilization (25°C)		3.7	3.7 3.1 3.0	10.5	9.5
Number of remaining <i>B.cereus</i> JFO13494 (CFU/mL)		<50	<50 <50 9.8x10 <sup>6</sup> 1.0x10 <sup>7</sup>	1.5x10 <sup>7</sup>	8.4x10 <sup>6</sup>
	<i>B.subtilis</i> var.niger	<50	<50 <50 2.9x10 <sup>7</sup> 3.3x10 <sup>7</sup>	4.0x10 <sup>7</sup>	3.1x10 <sup>7</sup>

sterilizing test with a diluted aqueous solution having an organic peracid's concentration of 3000 ppm.

sterilizing test with a starting aqueous solution.

sterilizing test with a starting aqueous solution.

\*: is Dequest 2010

\*\* the amounts of (A), (B) and Alkali pH adjusting agent are recited for 100 parts by weight of the total of (A) and (B).

← means the same as the left-sided term

EXHIBIT II

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Sir:

I, Noboru Matsuo, hereby declare as follows:

I am one of the co-inventors of the invention as described and claimed in the above-identified patent application.

I have carried out additional examples myself or under my direct supervision. Test procedures and results are shown below.

Example 5-9-a and Example 5-10-a

Example 5-9 and Example 5-10 were experimentally followed. Test conditions and test results are described in Table C, including additional conditions, hereto attached. An alkali agent and an acid agent were the alkaline pH adjusting agent and the acidic pH adjusting agent used in Example 1 of the instant application. The glycerin fatty acid ester had a fatty acid group having 8

carbon atoms and was the same as used in Example 1 of the instant application..

It is noted that Example 5-9-a is superior to Example 5-10-a by about 18 % in view of the reaction efficiency of production of the organic peracid.

The undersigned declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S. Code 1001 and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

By: Noboru Matsuo

Noboru Matsuo

Date: August 29, 2010

Attachment: Table C

Table C

	Ex5-9-a	Ex5-10-a
(A) glycerin fatty acid ester	5 (g) 0.0229 (mol)	8 (g) 0.0367 (mol)
(B) H <sub>2</sub> O <sub>2</sub>	0.29 (g) 0.0030 (mol)	0.29 (g) 0.0030 (mol)
(C) water	55 (g)	60 (g)
Alkali agent (initiator) + acid agent (terminating agent)	10 (g)	10 (g)
Total	70.29 (g)	78.29 (g)
peracid (C <sub>7</sub> H <sub>15</sub> COOOH: MW=160) concentration amount	4500 (ppm) 0.3163 (g) 0.00198 (mol)	5500 (ppm) 0.4306 (g) 0.00269 (mol)
efficiency of reaction peracid/(A)	0.0863	0.0733

## EXHIBIT III

Table 1

		Products of the invention									
		1-1	1-2	1-3	1-4	1-5	1-6	1-7	1-8	1-9	1-10
Compounding component	Ethylene glycol monoacetate	2g (0.0192)							3g (0.0288)		
	Ethylene glycol diacetate		2g (0.0137)						3g (0.0205)		
	Diacetin			2g (0.0114)						3g (0.0170)	
	(A) Triacetin				2g (0.0092)						
	Pentaerythritol tetraacetate					2g (0.0066)					
	Pentaacetyl-β-D-glucose						2g (0.0051)				
	Glycerine fatty acid ester							2g (0.0092)			
	Aqueous hydrogen peroxide (35 wt%)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)
	(B) Sodium percarbonate										
	Sodium perborate										
(A)/(B) molar ratio		0.65	0.47	0.39	0.31	0.22	0.17	0.31	0.98	0.70	0.58
pH(25°C)		4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.9	4.9	4.9
Organic peracid concentration (ppm)	Just after pH adjustment	6900	17000	10000	22900	4500	25800	2700	9800	21500	16100
	30 minutes after pH adjustment	6800	17000	9600	20300	4500	24100	2700	9600	21000	15700
	60 minutes after pH adjustment	6600	16800	9100	18600	4300	22000	2500	9300	19800	14200
	120 minutes after pH adjustment	6200	15200	8200	17500	3900	19800	2000	9000	18400	12300
Degree of remaining organic peracid (%)		89.9	89.4	82.0	76.4	86.7	76.7	74.1	91.8	85.6	76.4
H <sub>2</sub> O <sub>2</sub> /ester group molar ratio		1.53	1.07	1.28	1.07	1.11	1.15	3.20	1.02	0.717	0.865

Table 2

		Product of the invention									
		1-11	1-12	1-13	1-14	1-15	1-16	1-17	1-18	1-19	1-20
Compounding components	Ethylene glycol monoacetate					5g (0.0481)					
	Ethylene glycol diacetate						5g (0.0342)				
	Diacetone							5g (0.0284)			
	(A) Triacetone	3g (0.0138)							5g (0.0229)		
	Pentaerythritol tetraacetate		3g (0.0099)							5g (0.0164)	
	Pentaacetyl-β-D-glucose			3g (0.0077)							5g (0.0128)
	Glycerine fatty acid ester				3g (0.0138)						
	Aqueous hydrogen peroxide (35 wt%)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)
	(B) Sodium percarbonate										
	Sodium perborate										
(A)/(B) molar ratio		0.47	0.34	0.26	0.47	1.64	1.16	0.97	0.78	0.56	0.44
pH(25°C)		4.9	4.9	4.9	4.9	3.8	3.8	3.8	3.8	3.8	3.8
Organic peracid concentration (ppm)	Just after pH adjustment	25400	6800	28700	4100	12300	22000	23200	31700	9300	33500
	30 minutes after pH adjustment	24300	6100	27400	3900	11700	20500	22200	29900	8600	32000
	60 minutes after pH adjustment	23600	5700	26800	3700	10500	18800	21000	27600	7800	30600
	120 minutes after pH adjustment	20500	5000	24900	3500	10000	17800	20300	25800	7200	28100
Degree of remaining organic peracid (%)		80.7	73.5	86.8	85.4	81.3	80.9	87.5	81.4	77.4	83.9
H <sub>2</sub> O <sub>2</sub> /ester group molar ratio		0.710	0.742	0.764	2.13	0.611	0.430	0.518	0.428	0.448	0.459

Table 3

		Product of the invention										
		1-21	1-22	1-23	1-24	1-25	1-26	1-27	1-28	1-29	1-30	
Compounding components	Ethylene glycol monoacetate	2g (0.0192)										
	Ethylene glycol diacetate		2g (0.0137)						3g (0.0205)			
	Diacetin			2g (0.0114)						3g (0.0138)		
	(A) Triacetin				2g (0.0092)							
	Pentaerythritol tetraacetate					2g (0.0066)					3g (0.0077)	
	Pentaacetyl-β-D-glucose						2g (0.0051)					
	Glycerine fatty acid ester							2g (0.0092)				
	Aqueous hydrogen peroxide (35 wt%)											
	(B) Sodium hydrogen percarbonate	4.55g (0.0294)	4.55g (0.0294)	4.55g (0.0294)	4.55g (0.0294)	4.55g (0.0294)	4.55g (0.0294)	4.55g (0.0294)		5.00g (0.0294)	5.00g (0.0294)	
	Sodium perborate											
(A)/(B) molar ratio		0.65	0.47	0.39	0.31	0.22	0.17	0.31	0.70	0.47	0.26	
pH(25°C)		4.2	4.2	4.2	4.2	4.2	4.2	4.2	4.5	4.5	4.5	
Organic peracid concentration (ppm)	Just after pH adjustment	7000	17500	11500	23100	4600	26200	2900	23000	23600	26700	
	30 minutes after pH adjustment	6900	17000	9800	21500	4400	25100	2800	21600	21500	24300	
	60 minutes after pH adjustment	6800	16200	9300	19800	4300	22500	2600	20100	19700	23900	
	120 minutes after pH adjustment	6200	15000	8500	18600	3800	20000	2200	19200	18300	21300	
Degree of remaining organic peracid (%)		88.6	85.7	73.9	80.5	82.6	76.3	75.9	83.5	77.5	79.8	
H <sub>2</sub> O <sub>2</sub> /ester group molar ratio		1.53	1.07	1.28	1.07	1.11	1.15	3.20	0.717	0.710	0.764	

Table 6

		Product of the invention									
		2-1	2-2	2-3	2-4	2-5	2-6	2-7	2-8	2-9	2-10
Compounding ingredients	Ethylene glycol monoacetate	2g (0.0192)							3g (0.0288)		
	Ethylene glycol diacetate		2g (0.0137)								
	Diacetin			2g (0.0114)						5g (0.0229)	
	Triacetin				2g (0.0092)						
	Pentaerythritol tetraacetate					5g (0.0164)					
	Pentaacetyl- $\beta$ -D-glucose						2g (0.0051)				5g (0.0128)
	Glycerine fatty acid ester							5g (0.0229)			
	Aqueous hydrogen peracid (35 wt%)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)
	Sodium percarbonate										
	Sodium perborate										
(A)/(B) molar ratio		0.65	0.47	0.39	0.31	0.56	0.17	0.78	0.98	0.78	0.44
pH(25°C)		4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.9	3.8	3.8
Organic peracid concentration (ppm)		5000	5000	4000	4000	4000	4000	4000	4000	4000	4000
Number of remaining microorganisms (CFU/mL)	<i>Bacillus subtilis</i>	<50	<50	<50	<50	<50	<50	150	<50	<50	<50
	<i>Bacillus circulans</i>	<50	<50	<50	<50	<50	<50	200	<50	<50	<50
	<i>Aspergillus niger</i>	<50	<50	<50	<50	<50	<50	150	<50	<50	<50
H <sub>2</sub> O <sub>2</sub> /ester group molar ratio		1.53	1.07	1.28	1.065	0.448	1.15	1.28	1.02	0.428	0.459

Table 7

Compounding ingredients		Product of the invention									
		2-11	2-12	2-13	2-14	2-15	2-16	2-17	2-18	2-19	2-20
(A)	Ethylene glycol monoacetate	2g (0.0192)							3g (0.0288)		
	Ethylene glycol diacetate		2g (0.0137)								
	Diacetin			2g (0.0114)							
	Triacetin				2g (0.0092)					5g (0.0229)	
	Pentaerythritom tetraacetate					5g (0.0164)					
	Pentaacetyl - $\beta$ -D-glucose						2g (0.0051)				5g (0.0128)
	Glycerine fatty acid ester							5g (0.0229)			
	Aqueous hydrogen peroxide (35 wt%)										
	Sodium percarbonate	4.55g (0.0294)	4.55g (0.0294)	4.55g (0.0294)	4.55g (0.0294)	4.55g (0.0294)	4.55g (0.0294)	4.55g (0.0294)	5.00g (0.0294)	5.00g (0.0294)	5.00g (0.0294)
	Sodium perborate										
(A)/(B) molar ratio		0.65	0.47	0.39	0.31	0.56	0.17	0.78	0.98	0.78	0.44
pH(25°C)		4.2	4.2	4.2	4.2	3.9	4.2	3.9	4.5	3.9	3.9
Organic peracid concentration (ppm)		4000	4000	4000	4000	4000	4000	4000	4000	4000	4000
Number of remaining microorganisms (CFU/mL)	<i>Bacillus subtilis</i>	<50	<50	<50	<50	<50	<50	200	<50	<50	<50
	<i>Bacillus circulans</i>	<50	<50	<50	<50	<50	<50	200	<50	<50	<50
	<i>Aspergillus niger</i>	<50	<50	<50	<50	<50	<50	250	<50	<50	<50
percarbonate/ester group molar ratio		1.53	1.07	1.28	1.065	0.448	1.15	1.28	1.02	0.428	0.459





Table 13

Manufacturing conditions		Example											
		5-11	5-12	5-13	5-14	5-15	5-16	5-17	5-18	5-19	5-20		
Charging amount	(A)	Ethylene glycol diacetate 2g (0.0137)	3g (0.0205)	5g (0.0342)									
		Triacetin			2g (0.0092)	3g (0.0138)	5g (0.0229)						
		Pentaacetyl- $\beta$ -D-glucose						2g (0.0051)	3g (0.0077)	5g (0.0229)	8g (0.0367)		
		Glycerin fatty acid ester											
(B)		Aqueous hydrogen peroxide (35 wt%)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	2.86g (0.0294)	0.29g (0.0030)	0.29g (0.0030)		
		Sodium percarbonate											
(C)		Sodium perborate											
		Water	48g	48g	55g	48g	48g	48g	55g	55g	60g		
(A)/(B) molar ratio		0.47	0.70	1.16	0.31	0.47	0.78	0.17	0.26	7.63	12.23		
[(A)+(B)]/(C) ratio by weight		0.063	0.083	0.109	0.063	0.083	0.109	0.063	0.073	0.093	0.135		
pH in second step (25°C)		4.9	4.9	4.9	4.9	4.9	4.9	4.9	4.9	4.9	4.9		
Organic peracid concentration (ppm)	Just after second step	18300	22100	23600	22300	26100	33700	26900	29900	4500	5500		
	30 minutes after second step	16900	20700	20700	20500	23300	29500	23800	26700	4000	5100		
	60 minutes after second step	15800	18600	19200	18400	20100	27200	22400	23900	3800	4600		
Degree of remaining organic peracid (%)		86.9	84.2	81.4	82.5	77.0	80.7	83.3	79.9	84.4	86.3		
Degree of remaining hydrogen peroxide (%)		47.2	40.9	39.2	40.3	23.4	7.5	32.8	23.1	27.7	18.3		
H <sub>2</sub> O <sub>2</sub> /ester group molar ratio		1.07	0.717	0.430	1.07	0.710	0.428	1.15	0.764	0.131	0.082		

Table 14

		Example											
		5-21	5-22	5-23	5-24	5-25	5-26	5-27	5-28	5-29	5-30		
Manufacturing conditions	Charging amounts	(A)	Ethylene glycol diacetate	2g (0.0137)	3g (0.0205)	5g (0.0342)							
			Triacetin				2g (0.0092)	3g (0.0138)	5g (0.0229)				
			Pentaacetyl-β-D-glucose							2g (0.0051)	3g (0.0077)		
			Glycerin fatty acid ester								5g (0.0229)	8g (0.0367)	
	(B)	Aqueous hydrogen peroxide (35 wt-%)											
		Sodium percarbonate	4.55g (0.0294)	4.55g (0.0294)	4.55g (0.0294)	4.55g (0.0294)	4.55g (0.0294)	4.55g (0.0294)		5.00g (0.0294)	5.00g (0.0294)	0.5g (0.0029)	
		Sodium perborate											
	(C)	Water	55g	55g	55g	55g	55g	55g	55g	55g	55g		
	(A)/(B) molar ratio		0.47	0.70	1.16	0.31	0.47	0.78	0.17	0.26	7.90	12.66	
	[(A)+(B)]/(C) weight ratio		0.055	0.073	0.109	0.055	0.073	0.109	0.055	0.073	0.093	0.147	
pH in second step(25°C)		4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5		
Organic peracid concentration (ppm)	Just after second step	18500	22000	23000	22100	27300	33500	26400	30100	4500	5300		
	30 minutes after second step	17200	21000	22300	21600	25400	30800	24100	28300	4300	5300		
	60 minutes after second step	15800	19900	21500	20900	23200	29600	22500	25100	4100	5000		
Degree of remaining organic peracid (%)		85.4	90.5	93.5	94.6	85.0	88.4	85.2	83.4	91.1	94.3		
Degree of remaining hydrogen peracid (%)		46.2	38.6	35.6	37.2	20.8	6.9	30.9	21.1	25.9	17.4		
H <sub>2</sub> O <sub>2</sub> /ester group molar ratio		1.07	0.717	0.430	1.07	0.710	0.428	1.15	0.764	0.127	0.079		

